PROGRAM AND ABSTRACTS OF PAPERS CITRUS RESEARCH CONFERENCE

FRUIT AND VEGETABLE

CHEMISTRY LABORATORY

263 SOUTH CHESTER AVENUE

PASADENA, CALIFORNIA 91106

December 5, 1967

Western Utilization Research and Development Division
Agricultural Research Service
UNITED STATES DEPARTMENT OF AGRICULTURE

FOREWORD

This Citrus Research Conference is being held to bring to members of the citrus and allied industries in Southern California and Arizona the latest results of research on the chemistry, pharmacology, and technology of citrus fruits and their products carried on by the Utilization Research and Development Divisions of the Agricultural Research Service, U. S. Department of Agriculture. The following are participating in this year's conference.

- Western Utilization Research and Development Division:
  - Western Regional Research Laboratory (Division headquarters), 800 Buchanan Street, Albany, Calif. 94710
  - Fruit and Vegetable Chemistry Laboratory, 263 South Chester Avenue, Pasadena, Calif. 91106

- Southern Utilization Research and Development Division:
  - U. S. Fruit and Vegetable Products Laboratory, 600 Avenue S, N.W., Winter Haven, Florida 33882
  - Food Crops Utilization Research Laboratory 910 W. 6th Street, Weslaco, Texas 78596

PROGRAM

CITRUS RESEARCH CONFERENCE

Tuesday, December 5, 1967

STUDIES OF LIMONIN MONOLACTONE, THE NONBITTER PRECURSOR OF LIMONIN

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A long standing problem of juice manufacture from certain citrus fruits, particularly navel oranges, is the development of bitterness in the juice after extraction. Of special interest is the fact that, when first prepared, the juice is not bitter, but on standing bitterness gradually develops. It has been known for many years that the compound responsible for the bitter taste of navel orange juice is the triterpenoid limonin; however, all theories put forth to explain the delayed bitterness phenomenon have defied conclusive experimental proof. As a result, two fundamentally different theories, each based on indirect evidence, came into existence. They are commonly referred to as the precursor theory and the diffusion theory. The precursor theory holds that the fruit tissues contain a nonbitter precursor substance which diffuses into the juice where it is slowly converted into limonin. Conversion of the precursor into limonin is postulated to be catalyzed by acid and/or enzymes. The diffusion theory holds that initially limonin (not a precursor) is present in the juice as a constituent of the tissue fragments and because the limonin is not in solution the juice is not bitter. In time, it is thought, the limonin diffuses from the tissue fragments into solution whereupon the juice becomes bitter.

By taking a new approach based on chromatographic and electrophoretic techniques we have now directly demonstrated the presence of a nonbitter precursor in extracts of endocarp and albedo tissues of early season navel oranges and grapefruit. In addition, the nonbitter precursor has been identified as limonin monolactone (limonin with a hydroxy acid group in place of one lactone ring) by comparison with the synthetic compound and by acid-catalyzed conversion into limonin. In the intact fruit limonin monolactone is stable in the tissues (which are not bitter) because it is apparently not in direct contact with the acidic juice. When the fruit is converted into juice the broken tissues mix with the acidic juice and the nonbitter limonin monolactone, which is unstable under these conditions, is gradually converted into the intensely bitter compound limonin, thus accounting for the delayed development of bitterness. Limonin monolactone was not detected in late-season navel oranges or grapefruit nor was limonin present in the juices made from these fruit. These facts are in agreement with observations that late-season navel oranges frequently produce nonbitter juices.

Identification of limonin monolactone as the nonbitter precursor of limonin has necessitated studies of the various open-ring forms of limonin. Some of these forms of limonin (the dihydroxy diacid, the A-ring monolactone hydroxy acid, and the D-ring monolactone hydroxy acid) exist in equilibrium with each other under certain conditions. However, under moderately alkaline conditions only the diacid (disalt) exists, whereas under moderately acidic conditions only limonin exists. Also, the rate of conversion of one species into another is dependent on pH and temperature.

This new knowledge of the chemistry of the delayed bitterness phenomenon is proving to be very helpful in studies which may lead to techniques for producing juices free of limonin bitterness and in studies of the biochemistry of the limonoids.

REVIEW OF RECENT WORK ON LIMONOID BITTER PRINCIPLES

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Work conducted at the Pasadena laboratory during the past six years on the limonoid bitter principles occurring in citrus fruits will be reviewed. In 1961, only three limonoids of determined structure were known in citrus, largely the result of work by Ralph Higby at Sunkist and Oliver Emerson at the Western Regional Research Laboratory. These known compounds were limonin, obacunone, and nomilin. The present study has resulted in the isolation and structure determination of three additional limonoids from citrus, namely deacetylnomilin, deoxylimonin, and ichangin. The structure of two other limonoids, rutaevin and zapoterin, that occur in citrus relatives, also have been determined. These compounds form a reasonable biogenetics sequence arising from a tetracyclic triterpene leading to the formation of limonin. Zapoterin is a hydroxy derivative of obacunone and work leading to its structure determination will be discussed.

The botanical distribution of limonoids in citrus and its relatives has been explored. Limonoids have been found in Poncirus, Microcitrus, Fortunella, Eremocitrus, and Hesperethusa. Thus, all of the "true citrus fruit trees" according to Swingle's classification (with the exception of Clymenia which has not been examined because of lack of material) contain limonoids. Limonoids show in general a good correlation between oxidation state and their occurrence in the plant family Rutaceae. Limonoids of the same oxidation level occur in the same plant groups.

Techniques such as thin-layer chromatography, methods of detection, spectroscopic methods, etc. used in this study on limonoids have general usefulness in working with limonoids in the citrus processing industry.

THE OCCURRENCE OF CAROTENONES IN CITRUS

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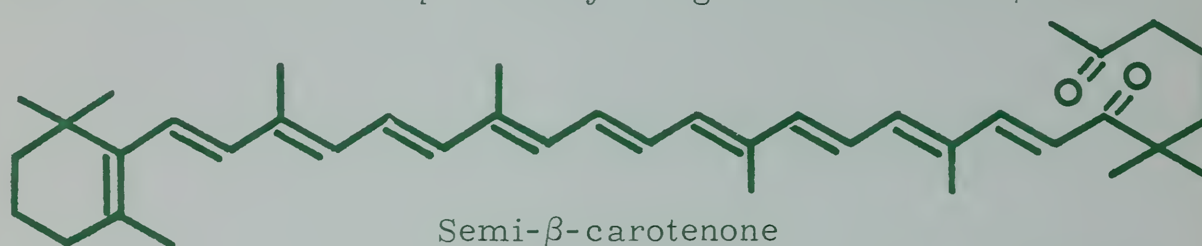
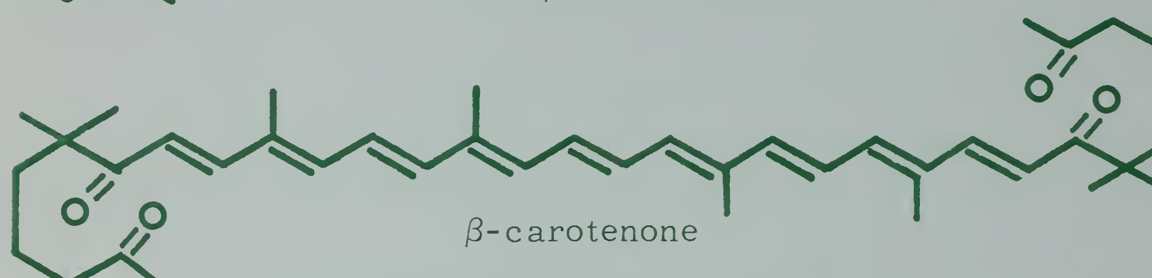
Semi-β-carotenone and β-carotenone have been known for a long time as partial oxidation products of β-carotene. By carefully controlled chromic acid oxidation of β-carotene, Kuhn and Brockmann in 1935 succeeded in isolating these two carotenones as cleavage products. This provided valuable confirmation to the previously assigned structure of β-carotene:



Semi-β-carotenone



β-carotenone

Since that time, although many other highly oxidized carotenoid pigments have been isolated from products such as citrus fruits, the naturally occurring semi-β and β-carotenones remained essentially undetected.

However, recent work at the Pasadena laboratory has shown that both carotenones occur in citrus fruits. Semi-β-carotenone was first isolated from the fruit of the orange jessamine (Murraya exotica Linn.). The fruit is bright red, and this rich color is due primarily to the presence of the carotenones. Semi-β-carotenone accounts for approximately 61 percent of the total carotenoids in the fruit; this amounts to approximately 180 mg. of total pigments (per kg. wet fruit) of which 110 mg. is due to semi-β-carotenone.

Subsequent to the initial isolation of semi-β-carotenone from Murraya, this carotenone was detected in large amounts in the fruit of the trifoliate limeberry (Triphasia trifolia (Burm. f.) P. Wils.). Accompanying the monocyclic ketone in this fruit there was a fair amount of a tetraketone which was structurally identified as β-carotenone. The fruit of the trifoliate limeberry is remarkably rich in its pigment content which amounted to

approximately 3 grams of total carotenoids per kilogram of wet fruit. Of this amount, the major portion (1.9 g. or 63 percent) is due to semi-β-carotenone. This fruit is undoubtedly one of the richest sources of carotenoids ever detected in nature.

In earlier studies conducted on the pigments involved in the "bronzing" of lemons, three carbonyl carotenoids were isolated. Of these, two were previously structurally identified as reticulataxanthin and the 3-hydroxy derivative of sintaxanthin. The third carotenoid has now been identified as semi-β-carotenone. A similar pattern of "bronzing" color development has been observed in mid-season Marsh grapefruit. Thus, it would not be surprising if on re-examination of grapefruit at the proper stage of ripeness, semi-β-carotenone is also detected in addition to the previously observed reticulataxanthin and 3-hydroxy-sintaxanthin. These findings should contribute to a better understanding of the pattern of degradation of the carotenoids in citrus fruits such as grapefruit and lemons and this will be discussed in detail.

JUICE CONCENTRATION BY REVERSE OSMOSIS

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Orange juice can be concentrated without adding heat or losing orange solids by a membrane process called reverse osmosis. Furthermore, most of the orange aroma is retained.

In reverse osmosis water diffuses under pressure through a semipermeable membrane. The success of the process for juice concentration depends upon the selectivity of the membrane and upon the rate of water removal. Selectivity is the ability of the membrane to pass water but retain dissolved solutes and suspended solids; it is determined by the chemical composition of the juice and of the membrane. For the cellulose acetate membranes now in use, fruit sugars and acids are well retained even at relatively high water permeation rates. In general, the aroma compounds of fruit juices are more difficult to retain but orange aroma is an exception. The oil soluble constituents of orange aroma do not permeate the membrane and are completely retained. Chromatographic analysis shows that a fraction of the small, water-soluble esters, aldehydes, and alcohols are lost through the membrane, particularly at high water removal rates. In spite of this, a concentrate prepared by reverse osmosis retains at least three times as much of the water-soluble aroma as does a concentrate made with cut-back juice and thus retains an excellent fresh orange flavor.

The permeation rate is not independent of selectivity, but depends on how the membrane is made. Tight membranes, which retain the water-soluble volatiles best, have low water removal rates, resulting in a longer product residence time and a more expensive process. Membranes made with higher permeation rates require less membrane surface, or lower pressures, and hence lower equipment costs, but can result in the loss of some volatiles or even dissolved solids. Sugar solutions of low osmotic pressure can be concentrated with excellent sugar retention at rates up to 30 gallons per square foot per day. As the osmotic pressure of the feed increases the permeation rate decreases according to the formula

$$\text{Permeation rate} = KA(P - \Delta\pi)$$

where P is the hydrostatic pressure applied to the feed, $\Delta\pi$ is (approximately) the osmotic pressure of the feed, A is the membrane surface area, and K is a permeation coefficient characteristic of the membrane. The osmotic

pressure of a four-fold orange juice concentrate is about 1300 p.s.i. Pressures necessary to produce practical permeation rates are therefore at least 1500 p.s.i. or higher. Suspended solids do not seriously interfere with reverse osmosis concentration.

There is no evidence at present that membrane life is a limiting factor in juice concentration by reverse osmosis. Periodic cleaning of the membrane equipment will be necessary, of course, and since cellulose acetate membranes are inexpensive, replacement of membranes in the dismantled equipment should not be costly.

A device has been designed - WURSTACK - which combines high strength with sanitary construction. This unit contains a high proportion of membrane surface to juice holdup - thus keeping residence time of the concentrate low. This design is presented and is recommended for R & D use at least.

BASIC AND APPLIED RESEARCH ON FOAM-MAT DRIED CITRUS JUICE CRYSTALS

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Basic research has concerned the study of nonenzymic browning associated with foam-mat dried citrus crystals during storage. Analytical results have indicated that for both orange and grapefruit similar products are formed during storage and in about the same quantities. Studies with a fructose base system and an ascorbic acid model system have indicated that some of the compounds found in stored citrus juice crystals probably derive from fructose and ascorbic acid. These findings complemented those of an earlier study and suggested possible precursors for storage products. In a study of correlation between ultraviolet absorption and heat treatment of chilled juice the two were found to be related when compared on a given sample. However, this correlation was not found between different samples.

Applied studies have concerned (1) the adaptations required in foam-mat drying of experimental orange concentrates varying widely in physical characteristics, (2) flavor evaluations of orange juice crystals containing different levels of flavor enhancement agents, (3) the effects of nonenzymic browning inhibitors such as SO_2 and carboxymethylcellulose on storage durability, and (4) the application of the foam-mat drying process to other subtropical fruits. For orange concentrates which were extremely different from the norm with regard to viscosity and pulp content, changes in foaming agents and foaming conditions were required. However, it was possible to develop foam-mat drying conditions yielding a satisfactory product for all concentrates. When concentrates containing high levels of peel oil were dried, approximately 60 to 75 percent of the oil was retained. Flavor evaluations indicated that untrained tasters prefer relatively high levels of peel oil. Storage studies showed storage durability of foam-mat dried orange crystals can be enhanced by SO_2, addition of certain types of carboxymethylcellulose, and use of preferred higher peel-oil levels. Satisfactory methods have been developed and dehydrated products produced from guava, pineapple, strawberry, orange, lemon and lime. These products were blended in some cases to provide additional new fruit drinks as well.

RECENT TRENDS IN THE CHEMISTRY OF THE DIHYDROCHALCONE SWEETENERS

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Three dihydrochalcone glycosides that are potentially useful as sweeteners have been discovered so far. These are naringin dihydrochalcone (I), neohesperidin dihydrochalcone (II) and hesperetin glucoside dihydrochalcone (III). These compounds are obtained from the citrus flavanones naringin, neohesperidin,

I: R_1 = H; R_2 = OH; R_3 = α-Rhamnosyl
II: R_1 = OH; R_2 = OMe; R_3 = α-Rhamnosyl
III: R_1 = OH; R_2 = OMe; R_3 = H

and hesperidin, respectively. Although each of these compounds has a pleasant, agreeable sweetness, a disadvantage is the fact that the sweetness starts too slowly and lingers too long. Various modifications have therefore been tried in order to see how these affect the quality of the sweetness. Most of these modifications have to do with adding or altering alkyl groups on the oxygen substituents at positions 3, 4, 2' and 6'. A summary of the available results will be given.

A second type of modification, which is much more difficult to carry out, is concerned with altering or blocking selected hydroxyl groups in the sugar part of the molecule in order to pinpoint the structural features of this part of the molecule that are required for sweetness. The modifications to be discussed involve alterations at the 6''-position of glucose.

A brief discussion will be given of some experiments directed towards the preparation of hesperetin glucoside dihydrochalcone by means of enzymic hydrolyses.

POTENTIAL MARKETS FOR DIHYDROCHALCONE SWEETENERS

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The consumption of noncaloric sweeteners has increased rapidly in the past five or six years. This expansion is likely to continue. The most important factor supporting this conclusion is the expected growth in sales of soft drinks, and the proportion of these products which is likely to be made with noncaloric sweeteners.

At the present time cyclamate and saccharin are the only two important noncaloric sweeteners in use throughout the world. In 1965 the U.S. production of cyclamate was 10.3 million pounds, and saccharin was 2.6 million pounds. Together they had a value of $11.7 million. It is estimated that by 1970, U.S. consumption will be about 21 million pounds of cyclamate and 4 million pounds of saccharin.

The dihydrochalcones are characterized by their pleasant sweetness and no bitter after-taste. The onset of sweetness is relatively slow but it is long-lasting. By combining these substances with cyclamate, it appears that a taste profile, very much like that of sucrose, can be obtained. The long-lasting sweetness of the dihydrochalcones might make them useful also in a number of products in which noncaloric sweeteners have not been used in the past, such as chewing gums, medicated troches or mouth washes.

If one-half (in terms of sweetening power) of cyclamate and of saccharin were to be replaced by naringin dihydrochalcone or hesperidin glucoside dihydrochalcone, it would represent an annual market for about 6.1 million pounds by 1970. Currently hesperidin and naringin sell for around $8 per pound, but if a large demand for them were to develop, the price might well drop to $1 per pound. Since about 1.5 pounds of the citrus flavanones would be required to make one pound of dihydrochalcones, this would represent sales of about $9 million per year for the citrus industry by 1970. In addition chemical manufacturers who convert the flavanones to dihydrochalcones would have sales of $11 million worth of the finished product.

THE TIME OF NARINGIN PRODUCTION IN GRAPEFRUIT

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(To be presented by Francis Griffith)

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The ovaries of pollinated grapefruit flowers were found to contain flavanones as one-third of their dry weight. Approximately the same relative amounts of the six major flavanone glycosides are found in both ovaries and mature grapefruit.

Exposure of the developing fruit and adjacent leaves to C-14 labeled carbon-dioxide was conducted at weekly intervals following fruit set. The relative amount of CO_2 fixation which occurred at different treatments was determined by measuring the C-14 activity in the sugar and organic acid fraction of the fruit. Activity was also followed in the naringin fraction. Carbon dioxide incorporation into the sugar-acids did not vary by greater than a factor of ten for those exposures conducted from early April to mid-June. The ratio of activity in the naringin fraction to that in the sugar-acid fraction reached a pronounced maximum in mid-April and then declined rapidly by mid-June to one-thousandth of the peak value. A uniformly low ratio of incorporation into the naringin fraction is maintained to early maturity (October).

Data thus indicate that the major period of naringin production is during the first month of fruit development and then declines rapidly. Cultural practices limiting naringin synthesis, if developed, would have optimum effectiveness during this period of maximum activity.

CHARACTERIZATION OF LEMON JUICE PRODUCTS

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Approximately 41 percent of the U.S. lemon crop is processed into juice and juice products. These products are sold on the basis of their acidity which is primarily citric acid. It is sometimes desirable to be able to characterize lemon juice in terms of constituents other than citric acid. To achieve this goal, methods of analysis were adapted to lemon juice for the measurement of total acidity as citric acid, total amino acids by formol titration, l-malic acid by optical rotation, and total phenolics by UV absorbance. Sixty-one samples of California-Arizona lemon juice concentrates were analyzed for these constituents and the data treated by a multiple regression approach. This yielded an equation whereby the citric acid content of a sample could be predicted on the basis of other constituents. Confidence limits were set at the 99 percent level which gave a maximum citric acid difference of 19.1 meq. /100 ml. This means that with natural juice, titrated citric acid values will be no more than 19.1 units away from the predicted values in 99 cases out of 100.

To further characterize lemon juice the individual amino acids were chromatographed on paper and passed through a recording densitometer. The pattern was fairly constant. Individual phenolics were also estimated by a paper chromatographic system and densitometer. The sum of the individual compounds closely followed the UV absorbance.

A study of the effects of fruit storage on the selected constituents showed that in one batch of lemons the l-malic acid dropped 64 percent in 15 weeks. The amino acid concentration increased by 34 percent, while the total phenolics and citric acid remained constant. In spite of these changes the citric acid values predicted by the multiple regression equation remained nearly constant.

The use of heavy extraction pressures by an *FMC extractor, as compared with light pressures, produced a small increase in the total amino acids and total phenolics in the extracted lemon juice. The other constituents were unchanged. Increased juice finishing pressures had the effect of increasing the pectin content but not the other constituents. The relationship between the predicted and measured citric acid values was not greatly changed.

*Use of commercial names does not imply endorsement by the Department of Agriculture.

Bottled lemon juice intended for room temperature storage at the retail level is commonly treated with chemical preservatives to prevent deterioration. A study of the effects of sulfur dioxide, sodium benzoate, and potassium sorbate preservatives on the previously mentioned analytical methods confirmed that there is no initial effect caused by these preservatives (except for the formol determination of amino acids, in which case the added SO_2 must be expelled by boiling 1 min.). During storage of the preserved juices at 5-35°C. for up to 17 weeks, small but statistically significant changes in composition were observed in some of the juice samples. For practical applications the changes did not affect the multiple regression approach.

There are other factors which could conceivably influence the composition of the juice such as variety, season, growing area, and cultural practices which have not yet been thoroughly investigated in this study. Furthermore, it would be impractical to even consider the many possible combinations of factors. However, limited investigations into some of the important ones indicate that the multiple regression approach may be applied to chemically characterize any commercial lemon juice product.

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GPO 975-177